भारतीय मानक
Indian Standard

IS 17450 : 2020

1, 3 फेंयलेनेडाईएमीन — विशिष्टि

1,3 Phenylenediamine — Specification

ICS 70.080.30

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

August 2020 **Price Group 3**

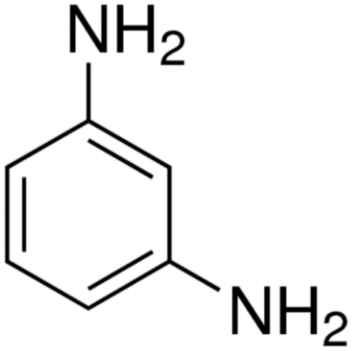
Dye Intermediates Sectional Committee, PCD 26

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

1,3-Phenylenediamine, also called 1,3-diaminobenzene, is an organic compound with the formula $C_6H_4(NH_2)_2$. It is an isomer of 1,2-Phenylenediamine and 1,4-Phenylenediamine.

1,3-Phenylenediamine is in aramid fibre manufacture, as a polymer additive, dye manufacturing, as a laboratory reagent, and in photography.



1,3 Phenylenediamine

CAS No.108-45-2

Molecular weight – 108.14

The composition of the Committee responsible for the formulation of this standard is given at Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding of numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

1,3 PHENYLENEDIAMINE — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and tests for 1,3 phenylenediamine

2 REFERENCES

The following standards contain provisions which through reference in this text constitute provisions of this standards. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification (*third revision*)
5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of cream solid material.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

4.2 Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Net mass of material;
d) Month and year of the manufacture; and
e) Batch or lot number.

4.3 BIS Certification Marking

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act,* 2016 and Rules and Regulations made there under. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in **4** of IS 5299.

5.2 Numbers of Tests

5.2.1 Tests for the determination of 1,3 Phenylenediamine, 1,2 Phenylenediamine and 1,4 Phenylenediamine shall be conducted on each of the individual samples.

5.2.2 Tests for the determination of remaining characteristics, namely, description, set point and moisture shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirement of 1,3 Phenylenediamine, 1,2 Phenylenediamine and 1,4 Phenylenediamine , if each of the individual test results satisfies the relevant requirements given in Table 1.

Table 1 Requirements for 1,3 Phenylenediamine

(*Clauses* 3.2, 5.3.1, 5.3.2 and 6.1)

Sl No.	**Characteristics**	**Requirement (On Dry Basis)**	**Method of Test, Ref to Annex**
(1)	(2)	(3)	(4)
i)	Purity by GC,percent by mass *Min.*	99.5	A
ii)	1,2 Phenylenediamine content, percent by mass *Max.*	0.10	A
iii)	1,4 Phenylenediamine content, percent by mass *Max.*	0.15	A
iv)	Set Point in °C *Min*	62	8 of IS 5299
v)	Moisture by Karl Fischer percent in mass *Max.*	0.5	10.2 of IS 5299

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics (*see* **5.2.2**) tested on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirements given in **3.1** and Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods referred in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i), (ii) *and* (iii)]

DETERMINATION OF 1, 3 PHENYLENEDIAMINE AND ITS IMPURITIESx

A-0 OUTLINE OF THE METHOD

The contents of 1,3 Phenyelenediamine and its impurity like 1,2 phenylenediamine and 1,4 phenyelediamine are determined by gas liquid chromatographic method. For details of test method, *see* IS 5299.

A-1 APPARATUS

Any gas chromatography capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used.

A-1.1 Objective

To determination purity of 1,3 phenyelenediamine by gas chromatography

A-2 REAGENT

a) Acetonitrle – AR grade;
b) 1,3,Phenylenediamine ;
c) 1,2,Phenylenediamine; and
d) 1,4,Phenylenediamine.

A-3 GC EQUIPMENT

Gas chromatography system with Flame ionization detector and auto injector.

A-4 GC PARAMETERS

Column : DB -17 30m * 0.53 mm, film thickness 1.50 µm or, equivalent column

Injector: split

Temperature : 300 °C

Split ratio : 5

Carrier gas : Nitrogen or helium with flow control mode pressure

Carrier gas pressure : 9.16 psi

Oven temperature:

Ramp Rate (°C)	Temperature (°C)	Hold Time (Min)
Initial	100	0.0
7.50°C/min	250	15.00

FID – Parameters

Hydrogen Flow : 40 ml/min

Air Flow : 400 ml/min

Temperature: 300 °C

Remarks

Other reagents or equipment may be used after testing for their suitability.

Aging of the column can influence the separation so that the conditions parameters like

Temperature and flow may have to be changed.

A-5 STANDARD PREPARATION

Weight accurately 1 gm of Standard 1,3, Phenylenediamine in 100 ml volumetric flask dissolve in acetone and make up the volume up to the mark with acetonitrile.

Weight accurately 0.5 gm standard 1,2, phenylenediamine in 100 ml volumetric flask dissolve in acetone and make up the volume up to the mark with acetonitrile.

Weight accurately 0.5 gm Standard 1,2, phenylenediamine in 100 ml volumetric flask dissolve in acetone and make up the volume up to the mark with acetonitrile.

A-6 SAMPLE PREPARATION

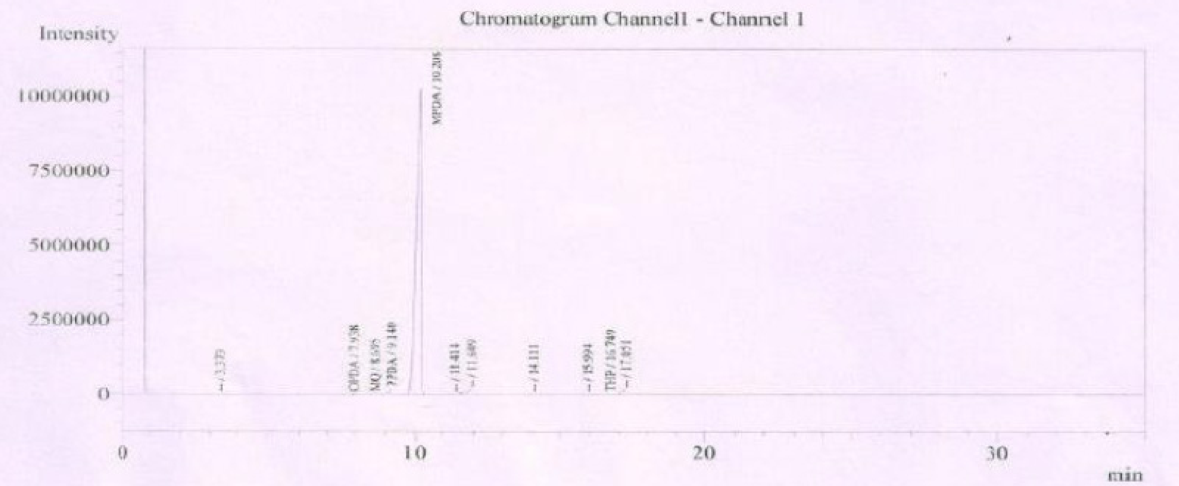
Weight accurately 1 gm of sample in 100 ml volumetric flask dissolve in acetone and make up the volume up to the mark with acetone.

A-7 INJECTION VOLUME

1.0 micro liter.

A-8 RETENTION TIME

[1] 1,2 Phenylenediamine : 7.93 min
[2] 1,4 Phenylenediamine : 9.14 min
[3] 1,3 Phenylenediamine : 10.21 min



Peak Table Channel 1 – Channel 1

Peak	Ret. Time	Area	Height	Area Prcent	Cmpd Name
1	3.373	1 694	361	0.001 5	–
2	7.938	2 442	391	0.002 1	OPDA
3	8.695	19 507	2 156	0.016 8	MQ
4	9.140	3 854	522	0.003 3	PPDA
5	10.208	115 949 822	10 299 929	99.919 8	MPDA
6	11.414	9 851	1 609	0.008 5	–
7	11.609	4 234	767	0.003 6	–
8	14.111	4 727	659	0.004 1	–
9	15.994	4 042	562	0.003 5	–
10	16.749	40 437	5 898	0.034 8	THP
11	17.051	2 326	392	0.002 0	–
Total		116 042 936	10 313 246	100.000 0	

ANNEX B

(*Foreword*)

COMMITTEE COMPOSITION

Dye Intermediates Sectional Committee, PCD 26

Organization	*Representative(s)*
ICT, Mumbai	Prof P. M. Bhate (***Chairman***)
Aarti Industries Limited, Mumbai	Shri Kirit H. Desai
Alkyl Amines Chemicals Limited, Mumbai	Shri Kirat Patel Shri S. V. Nikumbhe (*Alternate*)
Ankleshwar Research and Analytical Infrastructure Limited (ARAIL), Ankleshwar, Gujrat	Shri Mansukh H. Vekaria
Archroma, Thane	Dr Rajesh Ramamurthy Shri Hemant Mhadeshwar (*Alternate*)
Atul Limited, Valsad, Gujarat	Dr J. G. Desai Dr M. U. Rahman (*Alternate*)
BASF India Limited, Mumbai	Shri Uday Kulkarni
Central Revenue Control Laboratory (CRCL), New Delhi	Dr T. A. Sreenivasa Rao Shri Praful Dalal (*Alternate*)
Colourtex Industries Limited, Mumbai	Dr Pankaj Desai Shri Ismail Habibulla (*Alternate*)
Deepak Nitrite Limited, Vadodara	Shri Sailash Raval Dr Mangalya Kar (*Alternate* I) Dr J. K Astik (*Alternate* II)
Ecological and Toxicological Association of Dyes (ETAD), Vadodra	Dr Pariti Siva Rama Kumar
Gujarat Dyestuffs Manufacturers Association (GDMA), Ahmedabad	Shri R. S. Patel Shri Bansibhai Patal (*Alternate*)
Gujarat Pollution Control Board (GPCB), Ghandhinagar, Ahmedabad	Shri Y. A. Tai
Heubach Colour Private Limited, Mumbai	Shri J. I. Sevak Shri Vinod Madhukar (*Alternate*)
Indian Chemical Council, Mumbai	Shri P. S. Singh
Jay Chemical Industries, Ahmedabad	Shri Vilpesh Yadav Shrimati Maitri Vyas (*Alternate*)
Ministry of Chemicals & Fertilizers, Department of Chemicals & Petrochemicals, New Delhi	Shri Jasbir Singh
Ministry of Defence (Research and Development), New Delhi	Dr Vijay Tak
National Test House(er), Kolkata	Shri P. K. Chakraborty Shri Y. C. Nijhawan (*Alternate*)
Sudarshan Chemical Industries Limited, Pune	Dr Jaychandran M. Nair
The Bombay Textile Research Association	Dr Ashok N. Desai
The Dyestuffs Manufacturers Association of India (DMAI), Mumbai	Shri V. R. Kanetkar

Organization	*Representative(s)*
BIS Director General	SHRI N. K. BANSAL, SCIENTIST 'F' AND HEAD (PCD) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary

SHRI CHANDRAKESH SINGH
SCIENTIST 'D' (PCD), BIS

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This Indian Standard has been developed from Doc No.: PCD 26 (15671).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices: *Telephones*

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002 — 2323 7617, 2323 3841

Eastern : 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi KOLKATA 700054 — 2337 8499, 2337 8561, 2337 8626, 2337 9120

Northern : Plot No. 4-A, Sector 27-B, Madhya Marg CHANDIGARH 160019 — 265 0206, 265 0290

Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113 — 2254 1216, 2254 1442, 2254 2519, 2254 2315

Western : Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400093 — 2832 9295, 2832 7858, 2832 7891, 2832 7892

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Published by BIS, New Delhi